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EFFECT OF VARIATIONS IN SILICON AND IRON CONTENT ON EMBRITTLEMENT OF L-605 (HS-25)

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by Gary D. Sandrock, Richard L. Ashbrook, and John C. Freche Lewis Research Center Cleveland, Ohio

TECHNICAL PREPRINT prepared for National Metal Exposition and Congress sponsored by the American Society for Metals Detroit, Michigan, October 18-22, 1965



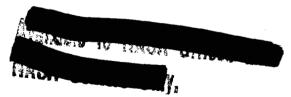
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION - WASHINGTON, D.C. - 1965

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Lewis Research Center
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ABSTRACT

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An investigation was conducted to study the effect of variations in silicon and iron content in L-605 (HS-25) on room temperature ductility and other mechanical properties after aging at 1600° F for various times up to 1000 hours. The silicon content of the alloy was investigated over the range 0.12 to 1.00 percent and the iron content over the range 0.16 to 3.24 percent. These ranges are generally within the manufacturer's specified nominal composition limits for these elements. This investigation showed that for low silicon contents (0.12 to 0.23 percent) the room temperature ductility of L-605 sheet aged for 1000 hours at 1600° F was improved over that of the high silicon (0.49 to 1:00 percent) heats as measured by tensile elongations. The former heats had elongations of 13 to 16 percent; the latter, 2 to 6 percent. Little apparent effect on room-temperature ductility after aging was observed as a result of the variation of iron content from 0.16 to 3.24 percent.

Aging at 1600° F reduced the room temperature ultimate tensile strength of L-605 for all the compositions investigated, and no overall relation between silicon content and ultimate tensile strength after aging was observed. Low-iron-content heats generally had higher ultimate tensile strengths after aging 1000 hours than did the high-iron-content heats. Hardness generally

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increased with aging time for all heats, although this effect was less pronounced for the low silicon heats.

Upon aging, precipitate formed preferentially at grain and twin boundaries as well as randomly in the matrix. The lower silicon heats had a lesser amount of precipitate after aging than did the high-silicon-content heats. Variations in iron content appeared to have little effect on microstructure after aging.

INTRODUCTION

The cobalt-base alloy L-605 (HS-25) has many elevated-temperature uses. Its mechanical and physical properties are summarized in Refs. 1 and 2. Because of its elevated-temperature strength, fabricability, and weldability, the alloy is of interest for aerospace applications. A potential application is its use in tubing and radiator components of advanced space power systems that are required to operate for mission times of thousands of hours. It has been observed (Ref. 3), however, that this alloy has a tendency to become brittle after long-time exposure to high temperatures. This property is obviously undesirable in engineering applications involving long-time exposure, particularly those subject to mechanical and thermal cycling.

Jenkins (Ref. 3) attributed the embrittlement of L-605 to the heavy precipitation of the intermetallic compound Co_2W during high-temperature exposure. Wlodek (Ref. 4) has suggested that the compound Co_2W is a stable Laves phase in this alloy. Since Co_2W is not an equilibrium phase in the Co-W binary system (Ref. 5), Wlodek has suggested that it is stabilized in L-605 by the silicon present. He contended that reduction in silicon content would lessen Laves phase precipitation and, in turn, reduce embrittlement.

Some data supporting this contention are shown in Refs. 4 and 6. It was also proposed in Ref. 4 that precipitation of carbides and the possible formation of hcp cobalt contributed to the embrittlement in L-605 and that the formation of hcp cobalt can be prevented by increasing the concentration of iron. Iron is believed to stabilize the fcc cobalt structure.

In view of the importance of retaining ductility in L-605, a program was undertaken at NASA Lewis Research Center to investigate the effect of wide variations of silicon and iron content, within the manufacturer's specifications, on the room-temperature mechanical properties of L-605 after aging. The room-temperature tensile strength and ductility, as well as the hardness of L-605 were determined after aging for various times up to 1000 hours at 1600° F. In addition, metallographic studies were made to gain further insight into the metallurgical mechanisms involved.

INVESTIGATIVE PROCEDURES

Material

Six special heats and two commercial heats, based on manufacturer's standard practice prior to 1964 (see CONCLUDING REMARKS), were obtained from the Union Carbide Stellite Company for use in this investigation. As wide a range of silicon and iron contents (generally within the manufacturer's nominal composition specifications) as was practically feasible was obtained. Table I shows the chemical compositions and grain sizes of the heats investigated. Complete chemical analyses were made by the supplier. The iron and silicon contents were also determined by an independent laboratory for all heats except heat 5. Silicon contents ranged from 0.12 to 1.00 weight per-

cent, and iron contents from 0.16 to 3.24 weight percent as determined by the independent laboratory. These analyses were used in the data plots presented in this paper. Average ASTM grain size varied from 4 to 6.

All material was hot-rolled to approximately 0.050 inch sheet and mill-annealed (2250°F, rapid-air cooled) by the supplier.

Tensile Tests

All tests were performed at room temperature. A standard "snap-on" type extensometer was used to obtain the load-strain curve to a strain of about 0.5 in./in. Strain rate was not directly controllable with this machine. Minor variations in strain rate that might have occurred would not be expected to greatly affect the tensile properties of this alloy at room temperature (Ref. 2). Figure 1 shows the tensile test specimen configuration used in this investigation. Machining was performed prior to aging. A one one-inch gage length was used in measuring elongation.

Hardness Tests

Hardness testing was done on tensile specimens near the shoulder of the specimen outside of the test length. A standard Rockwell machine, using the A scale (60 kg load-brale indentor), was employed. Prior to testing any thin oxide film present was removed either by grinding or by using a low-pressure dental gritblasting unit.

Aging Procedure

The specimens were aged at 1600° F in air. This temperature was selected because the greatest rate of embrittlement was observed (Ref. 4) with material that had been aged at this temperature. Specimens from all heats were aged 50, 200, and 1000 hours and then cooled in air to room temperature. Oxidation of the samples was slight, even after 1000 hours exposure at 1600° F.

Metallographic Studies

Longitudinal sections (in the rolling direction) of typical test specimens were taken for metallographic examination in the as-received (mill-annealed) condition and for each aging condition investigated. The as-received specimens were electrolytically etched in an HCl - 0.1-percent-H₂O₂ solution. Aged specimens were electrolytically etched in a boric acid-dilute H₂SO₄ solution. After etching, specimens were swabbed with NH₄OH to remove stains.

MECHANICAL PROPERTIES

Ductility

The average room temperature ductilities before aging and after aging at 1600° F for 50, 200, and 1000 hours are shown in Figure 2. For purposes of comparison the heats may roughly be divided into two groups, one of high (0.49 to 1.00 percent) and one of low (0.12 and 0.23 percent) silicon content. At all aging times the three heats with the low silicon content had substantially greater tensile ductility than the five high-silicon-content heats. After 50 hours aging, the low silicon heats had elongations that ranged from approximately 24 to 34 percent, while the elongations of the high silicon heats ranged between about 8 percent and 14 percent. After 200-hours aging the elongations of the low silicon heats ranged from approximately 17 to 32 percent as compared with only 3 to 7 percent for the high silicon heats. After 1000-hour aging, the low silicon heats still had a considerably greater ductility, 13 to 16 percent as against 2 to 6 percent for the high silicon heats. Four of the five high silicon heats had elongations ranging between approximately 2 and 3 percent.

Within each of the two groupings, the highest silicon-content heat (heat 6) did not always show the lowest ductility nor did the lowest siliconcontent heats (heats 7 and 8) always have the greatest ductility. Some crossover of the curves occurs so that the exact ranking of heats by ductility is not the same at each aging time. However, the trend toward improved ductility with lower silicon content is unmistakable, and a pronounced increase in ductility is clearly obtainable by reducing the silicon content to between 0.12 and 0.23 weight percent. This is illustrated more markedly in Figure 3, which presents the relation between ductility and silicon content for all heats after aging for 200 and 1000 hours, respectively. Varying the silicon content from 1 to 0.49 percent does not appear to have a pronounced effect on ductility; however, the ductility of the three heats with a silicon content of 0.23 percent or less was appreciably higher than that of the heats containing 0.49 to 1.00 percent silicon. The maximum silicon content for obtaining a specified ductility after aging has not been determined; however, reducing silicon content below 0.23 percent results in substantial improvements.

When heats within a narrow composition range of iron (0.16 to 0.57 percent) are considered, ductility after 200- and 1000-hours aging is still seen to increase with decreasing silicon content. On the other hand, when heats within narrow composition ranges of silicon (0.12 to 0.23 and 0.49 to 0.60 percent) are considered, no consistent trend of increasing ductility with increasing iron content is observed. Little apparent effect on postaging ductility was observed as a result of variations in iron content from 0.16 to 3.24 percent.

Tensile Strength

The average ultimate tensile strengths are plotted in Figure 4 as functions of 1600° F aging time for all heats investigated. In the asreceived (mill-annealed) condition all eight heats investigated had ultimate tensile strengths greater than the 130,000 psi minimum given by the Aerospace Materials Specification for L-605 (AMS 5537B, Ref. 7). It is also interesting to note that all six special heats investigated had ultimate tensile strengths in the mill-annealed condition greater than the two commercial heats (heats 4 and 5). This indicates that the tensile strength of the mill-annealed sheet is not adversely affected by reductions in silicon content. Of course, some of the improvement in tensile strength may be due to the somewhat smaller grain size of the special heats (see Table I).

Room-temperature ultimate tensile strength generally decreased with aging time. The decrease was most pronounced after the first 50 hours. With aging time greater than 50 hours, the decrease in ultimate tensile strength continued, but the decrease was generally less marked than after the first 50 hours. Heats 4 and 5 tended to regain part of their original strength after 200 hours of aging, but after 1000 hours these two heats again showed a decrease in ultimate tensile strength.

As a result of the general loss in ultimate tensile strength with aging time, the ultimate tensile strengths of some heats fell below the AMS minimum of 130,000 psi for L-605 after 1000-hours aging. For all of the aging conditions investigated the tensile strength of heat 4 was less than the AMS minimum.

There does not appear to be any clear-cut relation between silicon content and ultimate tensile strength after aging 1000 hours; however, the

low-iron-content heats generally had higher ultimate tensile strengths after aging for 1000 hours than did the high iron-content heats. It is interesting that after aging 1000 hours the tensile strengths of the low silicon heats investigated were close to (either slightly above or below) the AMS specifications and generally superior to the two commercial heats.

The 0.2 percent offset yield strengths are also plotted against aging time in Fig. 4. There is a general tendency for the high silicon heats to have higher yield strengths than those of the lower silicon heats after aging. After 50-hour aging, there is relatively little change in yield strength with increased aging time for any of the heats. The highest-silicon-content heat (heat 6) had the highest yield strength in the mill-annealed condition. Its ultimate tensile strength (Fig. 4) was similarly high compared with the other heats.

Hardness

Fig. 5 shows room-temperature hardness as a function of aging time for all heats investigated. All data points indicated represent the average of five hardness readings. There is a general increase in hardness with aging time for all heats. This increase in hardness is associated with increased precipitation. In general, after aging, the hardness of the higher-silicon-content heats increased more than the lower-silicon-content heats. After aging for 1000 hours, for example, the three lowest silicon content heats (heats 1, 7, and 8) had the lowest hardnesses. It is evident from metallographic studies that substantially less precipitation occurs during aging in the low silicon than in the high silicon heats. This would

explain the lower hardness of the low-silicon-content heats and tend to substantiate the contention that Laves phase precipitation is reduced by lowering silicon content.

Stress Rupture

Fig. 6(a) shows the stress-rupture life at 1600° F and 17,500 psi stress for all heats investigated, except heat 5, as a function of silicon content. There does not appear to be any clear-cut relation between stress-rupture life and silicon content. However, the relatively low stress-rupture lives obtained with the two lowest-silicon-content heats suggest that the stress-rupture life may possibly be adversely affected at very low silicon contents.

Fig. 6(b) similarly shows the effect of iron content on the stress-rupture life for the same test conditions. There appears to be a trend of decreasing stress-rupture life with increasing iron content, although the trend is not well defined.

MICROSTRUCTURAL CONSIDERATIONS

Effect of Aging on Microstructure of Intermediate
Silicon and Iron Content Heat

Fig. 7 shows the microstructure of a commercial heat of L-605 (heat 4) that was made by the manufacturer's standard practice for this alloy prior to 1964. Fig. 7(a) shows the as-received mill-annealed material. The microstructure consists largely of a solid solution of fcc cobalt. Twinning is evident in the grains. A few carbides that were not taken into solution during the annealing treatment are scattered throughout the matrix. After aging 50 hours at 1600° F precipitation of a second phase is evident (Fig. 7(b)).

Initial nucleation is particularly pronounced along grain and twin boundaries, although some also occurs randomly within the grains. After aging 200 hours, the amount of precipitate, both in the grain boundaries and within grains, was greatly increased (Fig. 7(c)). It should also be noted that many of the randomly located precipitate particles are rodlike in shape and tend to be lined up in preferred crystallographic directions. After aging for 1000 hours, the precipitate particles grew and possibly additional precipitates were formed (Fig. 7(d)). The microstructure is similar to that observed (Ref. 4) by Wlodek who identified the Laves phase, Co₂W, in aged heats of similar silicon content. The structure shown in Fig. 7(d) is quite brittle, only about 3.5 percent tensile elongation having been measured at fracture with tensile specimens aged 1000 hours at 1600° F.

Effect of Silicon Content on Microstructure After Aging

To observe the effects of silicon content on the microstructure of L-605, photomicrographs are compared in Fig. 8 for heats of varying silicon content (0.12 to 0.73 percent) but relatively constant iron content (0.16 to 0.57 percent) after aging 1000 hours. The two highest silicon heats 0.73 and 0.60 percent silicon (Figs. 8(a) and (b)), respectively, both show heavy precipitation; however, with a reduction to 0.23 percent silicon (Fig. 8(c)) a definite decrease in the amount of precipitate is evident. Fig. 8(d) shows the microstructure of the lowest silicon heat (0.12 percent). The amount of precipitate is less than that in any of the other heats. The preceding comparisons were also made with material aged 50 and 200 hours with similar results. It is evident from these metallographic studies that a reduction in silicon content greatly reduces precipitation after aging. This reduction in the

amount of precipitate is believed to be associated with increased ductility in L-605 after aging.

It should be noted that X-ray diffraction analysis of the 0.23 and 0.60 percent silicon heats (heats 1 and 2) conducted by Wlodek (Ref. 6) indicated only traces of Co₂W (Laves phase) present in the lower silicon heat and substantially greater amounts in the higher silicon heat. Although precipitation of the Laves phase has been greatly reduced in the lower silicon-content heats, it is evident that appreciable quantities of precipitate are still present after aging. Many of these precipitate particles are probably carbides of the M₆C type as identified by Wlodek in the 0.23 silicon heat (Ref. 6). The presence of such carbides particularly along grain boundaries could contribute to the degree of embrittlement that still exists, even in the low silicon compositions.

Effect of Iron Content on Microstructure After Aging

Figure 9 shows the microstructure of heat 7, which is low in iron (0.16 percent), and heat 8, which is high in iron (3.06 percent), after aging for 1000 hours. Both heats have the same silicon content (0.12 percent). The microstructures of both heats appear to be quite similar. This similarity was also observed in samples of both these heats after aging for 50 and 200 hours. Thus, iron does not appear to have a pronounced effect on the amount and nature of the precipitate, at least at low silicon contents. It is noteworthy that the ductility of the low silicon, low iron composition (heat 7) is substantially less at intermediate aging times than that of the low silicon, high iron composition (heat 8). The reason for this difference was

not apparent from a comparison of the microstructures.

Fracture Modes

Visual observation of the fracture surfaces of specimens broken in tensile tests shows a different fracture mechanism occurring in specimens that were aged as opposed to those in the mill-annealed condition. Fig. 10 shows a typical example of this difference. The upper part of the figure shows the fracture surface of a tensile specimen from heat 3 aged 1000 hours at 1600° F. A jagged fracture surface is evident. The lower part of the figure shows the fracture surface in a mill-annealed or unaged specimen from the same heat. The surface is fibrous in nature, and fracture appears to be typical of the shear failures encountered in ductile materials. The fracture surface in the aged specimen was at 90° to the tensile axis, while the fracture surface in the mill-annealed specimen was at an angle of about 60° to the tensile axis. Another aspect to be noted from the figure is the difference in width of each section. A much greater reduction in area occurred with the more ductile mill-annealed specimen. The types of fractures shown in Fig. 10 are generally typical of those encountered in all heats in the mill annealed and aged conditions.

It was observed at higher magnifications, however, that the amount of silicon had some influence on the fracture mode of specimens after aging. Figure 11(a) shows (at X250) the fracture of a tensile bar of heat 3 aged 1000 hours (same conditions as shown in Fig. 10, top). Heat 3 is a high silicon (0.73 percent) heat that had an elongation of only 2.7 percent after 1000 hours aging. Fracture was almost entirely intergranular, apparently due to heavy precipitation particularly along grain boundaries. Fig. 11(b)

shows a typical tensile fracture incurred in a specimen of heat 7, a low silicon (0.12 percent) heat that had an average elongation of 12.6 percent after 1000 hours aging. In this case, although some intergranular fracture occurred, a substantial degree of transgranular fracture took place as well. Grain boundaries can be traced across the fracture as indicated by the arrows in Fig. 11(b). Substantially less precipitation occurred in the specimen from heat 7. The amount of precipitate thus appears to have a noticeable effect on the nature of the fracture encountered.

CONCLUDING REMARKS

Recently the Union Carbide Corporation (Stellite Division) introduced a new melting practice in the production of L-605 (HS-25); as a result the silicon content has been substantially lowered. Data obtained by the manufacturer (Ref. 8) for heats made by this practice are given in Table II. These data show that low silicon content results in elongations of 6.4 to 17 percent after 1000-hour aging at 1600° F, as compared with 3 to 5 percent with sheet similarly aged and made by the practice employed prior to 1964. The tensile strengths reported in Ref. 8 for the heats listed in Table II are somewhat lower than those obtained in the present investigation with low silicon heats.

In summary, it appears that improved post-aging ductility in L-605 sheet can also be obtained in commercial practice by melting procedures that provide a low silicon content.

SUMMARY OF RESULTS

The following results were obtained from an investigation to determine the effect of silicon and iron content on the room-temperature ductility

and other mechanical properties of L-605 after aging at 1600° F for various times up to 1000 hours:

- 1. Reductions in silicon content increased ductility for all aging times (50, 200, and 1000 hours). For example, after aging 1000 hours at 1600° F the tensile elongations for the low silicon (0.12 to 0.23 percent) heats ranged from approximately 13 to 16 percent compared with 2 to 6 percent for the high silicon (0.49 to 1.00 percent) heats.
- 2. Little apparent effect on post-aging ductility was observed as a result of variations of iron content from 0.16 to 3.24 percent.
- 3. The ultimate tensile strength of all heats generally decreased with aging time. There was no overall relation between silicon content and ultimate tensile strength after aging. The low-iron-content heats generally had higher ultimate tensile strengths after aging for 1000 hours than did the high-iron-content heats.
- 4. Hardness generally increased with aging time for all heats investigated. In general, the hardness of the high silicon heats increased more than the hardness of the low silicon heats. For example, after 1000-hour aging at 1600° F the low silicon heats had hardnesses ranging from 62 to 66 Rockwell hardness (A scale) as against 68 to 70 for the high silicon heats.
- 5. The fracture surface in as-received mill-annealed specimens had a fibrous appearance typical of that observed in ductile materials; whereas, the fracture surface of aged specimens, regardless of silicon or iron content, was jagged in nature. However, in the aged condition, fracture in high silicon heats was predominantly intergranular whereas in low silicon heats there was a substantial degree of transgranular fracture as well.

6. Aging at 1600° F resulted in pronounced precipitation both preferentially along grain and twin boundaries and randomly throughout the matrix. The low-silicon-content heats had a lesser amount of precipitate after aging than did the high-silicon-content heats. Variations in iron content appeared to have little effect on the microstructure after aging.

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- 8. Private Communication, Union Carbide (Stellite Division) data.

TABLE 1. - CHEMICAL COMPOSITIONS AND GRAIN SIZES OF HEATS INVESTIGATED

ASTM average grain size				0004 4000
	S		1	0.019 .013 .010 .010 .010
	Ъ	Manufacturer's specifications	-	0.001 .005 .012 .014 .004
	Ço		Balance	Balance 0.001 Balance .005 Balance .012 Balance .014 Balance .004 Balance .004 Balance .004
	Mn		1/2	10.40 1.52 10.12 1.40 10.20 1.44 10.06 1.52 9.90 1.37 10.14 1.11 10.42 1.10
lier	Ni		9/11	10.40 1.52 10.12 1.40 10.20 1.44 10.06 1.52 9.90 1.37 10.14 1.11 10.42 1.10
Determined by supplier	ນ		0.05/0.15	0.13 .11 .08 .09 .10
Determir	W		14/16	15.02 15.42 15.66 14.62 15.05 14.75 14.32
	$c_{\mathbf{r}}$		19/21	0.60 20.02 .58 20.16 .20 20.14 1.67 20.07 1.85 20.09 2.90 20.79 9/.23 20.41 2.88 20.06
	Fe		a ₃	2.90/
	Si		aı	0.20 .50 .96 .63 1.33/1.12 .04/.11
er- l by le- ent or-	F.e		:	0.23 0.57 .60 .49 .73 .24 .55 1.60 5 1.60 2 3.24 2 3.06
Deter- mined by inde- pendent labor- tory	Si		;	0.23 0.57 .60 .49 .73 .24 .55 1.60 .100 3.24 .12 3.06
Heat Deter- number mined by inde- pendent labor- tory				1024 G 2007 8

 $^{\rm 2}{\rm Maximum}$ according to manufacturer's specifications (Ref. 1). $^{\rm 2}{\rm Manufacturer}$'s standard practice prior to 1964.

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TABLE II. - PRELIMINARY DATA ON RECENT UNION CARBIDE PROCEDURE FOR PRODUCTION

OF L-605 (HS-25) SHEET (REF. 8)

Heat	Composition			Tensile properties ^a		
number	Si	С	Fe	Yield 0.2 percent offset, psi	Ulti- mate, psi	Elon- gation, percent
64-590 64-627 L4-1677S L4-1696S	0.02 .03 .04 .10	.04 .09	1.86 1.98 1.52 2.27	67,000 65,000	123,000 121,000 118,000 108,000	16 17 11 6.4

^aAged 1000 hr. at 1600° F; average of 2 to 4 tests.

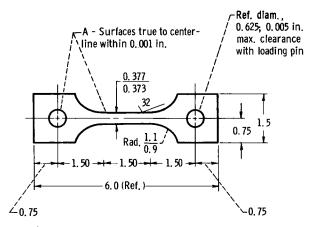


Fig. 1. – Tensile test specimen. (Unless otherwise noted, dimensions may vary ± 0.01 in.)

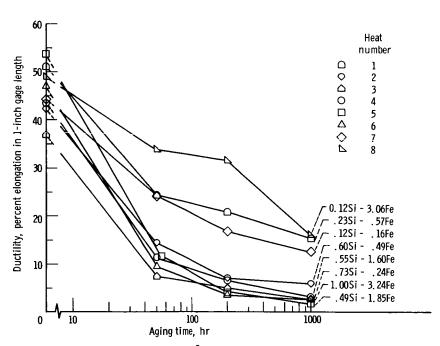


Fig. 2. - Effect of aging time at $1600^{\rm O}\,{\rm F}$ on average room temperature ductility.

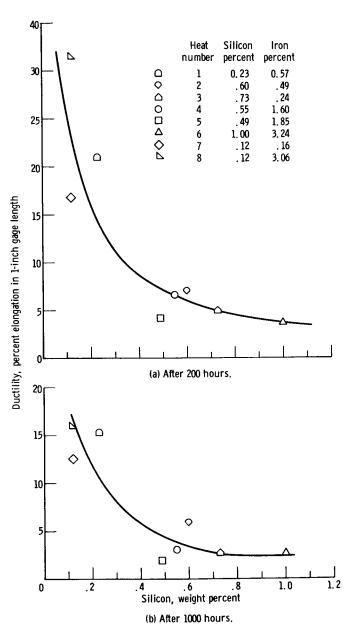


Fig. 3. - Effect of silicon content on average room-temperature ductility after aging at 1600° F.

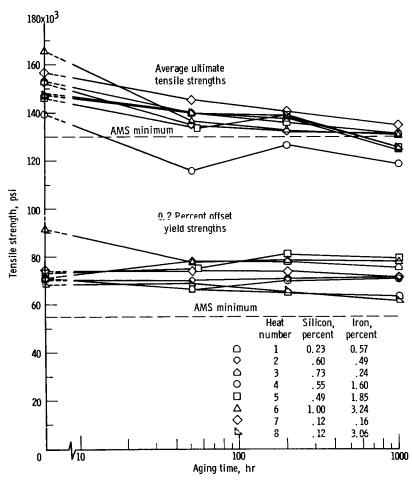


Fig. 4. - Effect of aging time at 1600° F on average room-temperature tensile strength.

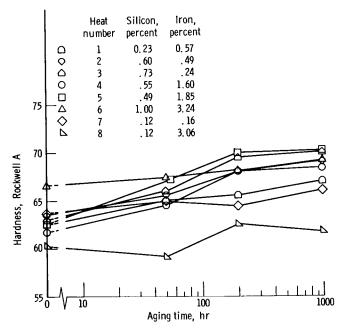


Fig. 5. – Effect of aging time at $1600^{\circ}\ \text{F}$ on average room-temperature hardness.

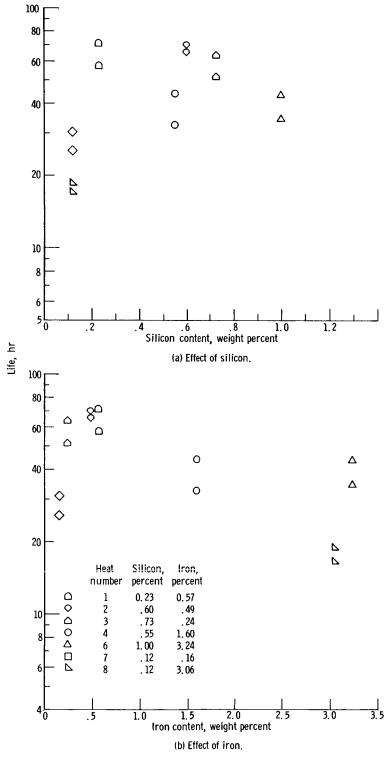


Fig. 6. - Stress-rupture life of mill-annealed L-605 sheet at 1600° F and 17,500 psi as a function of silicon and iron content.

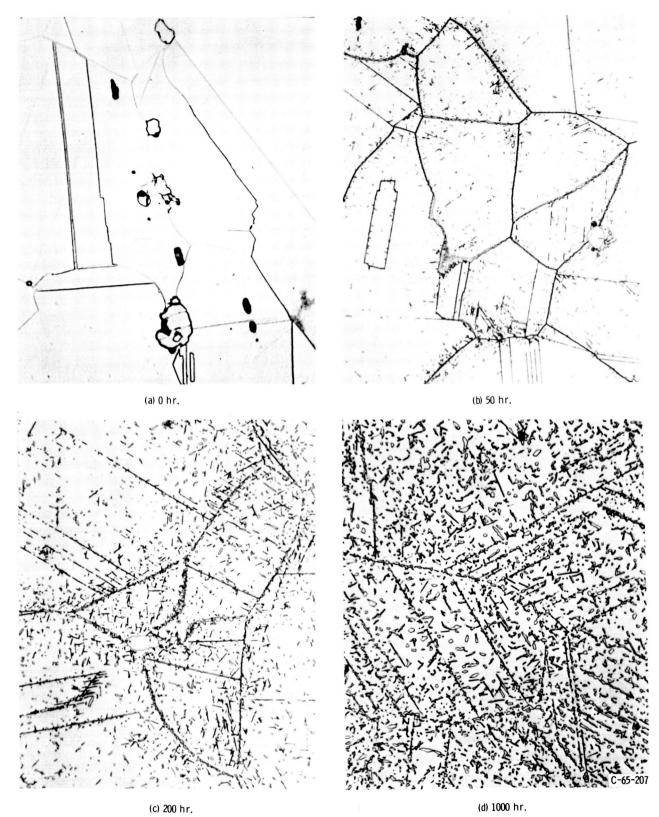


Figure 7. - Effect of aging time at 1600° F on microstructure of intermediate silicon and iron composition (0.55 Si - 1.60 Fe, heat 4). X750.

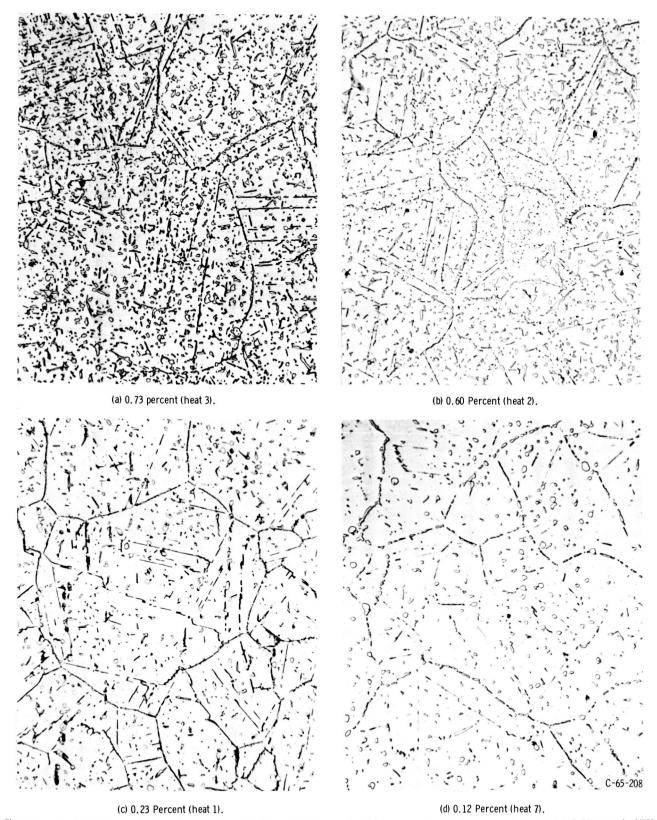
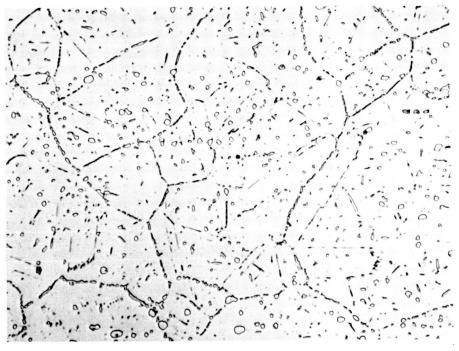
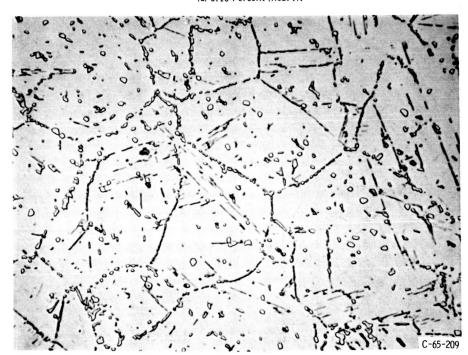


Figure 8. - Effect of silicon content on microstructure of L-605 aged 1000 hours at 1600° F; iron content for heats ranged from 0.16 to 0.57 percent. X750.



(a) 0.16 Percent (heat 7).



(b) 3.06 Percent (heat 8).

Figure 9. - Effect of iron content on microstructure for two heats of L-605 aged 1000 hours at 1600° F; silicon content identical for both heats (0.12 percent). X750.

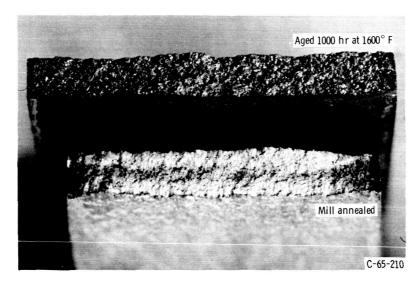
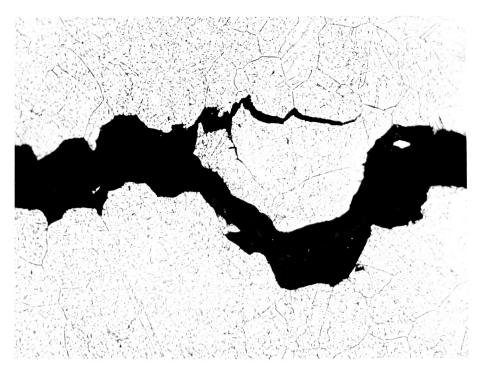
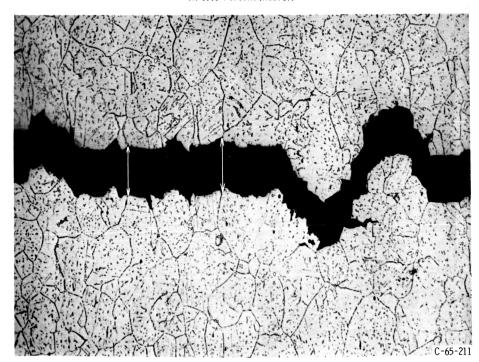


Figure 10. – Typical tensile fractures before and after aging (heat 3). $\,$ X10.



(a) 0.73 Percent (heat 3).



(b) 0.12 Percent (heat 7).

Figure 11. - Effect of silicon content on tensile fracture of L-605 sheet aged 1000 hours at 1600° F. X250. (Arrows indicate some grain boundaries that can be traced across the fracture.)